

# PATENT SPECIFICATION

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## (54) PROCESS FOR DECOLOURIZING POLYETHYLENE POLYAMINES

(71) We, TOYO SODA MANUFACTURING CO. LTD., a Japanese Company of No. 4560 Oaza-tonda, Shin-nanyo-shi, Yamaguchi-ken, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for decolourizing polyethylene polyamines. Throughout the description and the claims of this specification the term "polyethylene polyamine" is intended to include ethylene diamine, for the sake of convenience. More specifically the polyethylene polyamines to which the invention relates are compounds containing the following group:



- where n is an integer of 1 to 5, for example, compounds of the general formula



where n=1 to 5. Preferably the polyethylene polyamines are linear chain compounds having from 4 to 10 carbon atoms, although cyclic compounds, e.g. piperazine, can be used.

It is well known that polyethylene polyamines become discoloured during preparation or during storage in air. The higher polyethylene polyamines are particularly susceptible. The commercial value of the discoloured polyethylene polyamines is less than the colourless product.

Various processes have been devised to decolourize these compounds, for example, zinc metal may be added to the discoloured polyethylene polyamines followed by heating. Alternatively zinc metal and water may be added and the mixture heated or a metal component selected from zinc, aluminium and tin, and an alkaline component may be added and the mixture heated.

In all these conventional processes it can

be seen that a metal component is employed and consequently they all suffer from the common feature that if large pieces of the metal are employed it takes a fairly long time for the decolourization. Also practical operation of these methods is quite difficult. Accordingly, it is necessary to employ powdered metal for speed.

However, when powdered metal is employed the mixture needs to be stirred and if a mechanical stirrer is used an effective seal is necessary. Moreover, it is necessary to recover the powdered metal from the resulting mixture for re-use after terminating the decolourizing process and special apparatus is required for the separation.

Accordingly, the inventors have attempted to find a process which overcomes these difficulties.

The object of the invention is to provide a new process for decolourizing polyethylene polyamines.

According to the invention there is provided a process for decolourizing discoloured polyethylene polyamines (as defined herein) which comprises distilling the discoloured polyethylene polyamine in the presence of a polyethylene polyamine hydrochloride.

The decolourizing mechanism is not yet clearly understood.

The following examples clearly show that the decolourizing effect is extremely high.

The polyethylene polyamine hydrochlorides are prepared by reacting a polyethylene polyamine and hydrochloric acid.

The amount of the polyethylene polyamine hydrochloride employed depends on the degree of discolouration of the polyethylene polyamine. Usually the amount of polyethylene polyamine hydrochloride used is 0.1 to 10 parts by weight, preferably 0.5 to 5 parts by weight of the HCl component to 100 parts by weight of the polyethylene polyamine.

When the amount of the hydrochloride used is less than this a satisfactory decolourization effect is usually not obtained. On the other hand, when more hydrochloride is used no

having a concentration of 0.5 to 10 weight percent which were present in the mixture due to incomplete neutralization of the reaction product with NaOH. Thus, various polyethylene polyamines having the following hues and high boiling points were obtained.

TABLE 3

	Hue Number (Pt—Co hue standard)
10 Polyethylene polyamines	less than 10
Diethylene triamine	less than 10
N-2-Aminoethyl piperazine	less than 30
Triethylene tetramine	less than 100
15 Tetraethylene pentamine	less than 300
Pentaethylene hexamine	

## Comparison Example

A reaction product containing polyethylene polyamines was produced under the same reaction conditions as in Example 3. The said reaction product was treated in the absence of the hydrochlorides of the polyethylene polyamines throughout various steps from the neutralization step to the vacuum fractional distillation step and thus, the polyethylene polyamines having the following hues and high boiling points were produced.

TABLE 4

	Hue Number (Pt—Co hue standard)
30 Polyethylene polyamines	40
Diethylene triamine	50
N-2-Aminoethyl Piperazine	300
Triethylene tetramine	700
35 Tetraethylene pentamine	Over 1000
Pentaethylene hexamine	

## WHAT WE CLAIM IS:—

1. A process for decolourizing discoloured polyethylene polyamines (as defined herein) which comprises distilling the discoloured polyethylene polyamine in the presence of a polyethylene polyamine hydrochloride.

2. A process as claimed in claim 1 wherein the polyethylene polyamine hydrochloride is

formed by adding hydrochloric acid in less than the stoichiometric amount to the polyethylene polyamine prior to the distillation.

3. A process as claimed in claim 1 or claim 2 wherein the polyethylene polyamine comprises a linear chain polyethylene polyamine having from 4 to 10 carbon atoms.

4. A process as claimed in claim 1 or claim 2 wherein the polyethylene polyamine comprises one or more of the following: piperazine, N-2-aminoethyl piperazine, diethylene triamine, triethylene tetramine and pentaethylene hexamine.

5. A process as claimed in any preceding claim wherein the proportion of the polyethylene polyamine hydrochloride used is 0.1 to 10 parts by weight calculated as the hydrochloric acid component to 100 parts by weight of the polyethylene polyamine.

6. A process as claimed in any preceding claim wherein the proportion of the polyethylene polyamine hydrochloride used is 0.5 to 5 parts by weight calculated as the hydrochloric acid component to 100 parts by weight of the polyethylene polyamine.

7. A process as claimed in any preceding claim wherein the distillation temperature is from 80°C to 230°C and wherein the distillation takes place at a pressure of from 100 to 0.1 mmHg.

8. A process as claimed in any preceding claim wherein 35% aqueous hydrochloric acid solution is added to the polyethylene polyamine before distillation.

9. A process as claimed in claim 1 substantially as described herein with reference to the Examples.

10. Decolourized polyethylene polyamines produced by a process as claimed in any preceding claim.

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